

**TRANSMITTAL LETTTER TO THE UNITED STATES  
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CONCERNING A FILING UNDER 35 U.S.C. 371**

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see 37 CFR 1.55)

09/744237

INTERNATIONAL APPLICATION NO.  
PCT/FR99/01975

INTERNATIONAL FILING DATE  
August 12, 1999

PRIORITY DATE CLAIMED  
September 2, 1998

**TITLE OF INVENTION**  
**PROCESS FOR CONTROLLING THE PARTICLE SIZE IN A BAYER CIRCUIT PRECIPITATION SYSTEM, INCLUDING**  
**AN AGGLOMERATION PHASE**

**APPLICANT(S) FOR DO/EO/US**

Benoit Cristol

Applicant herewith submits to the United States Designated Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19<sup>th</sup> month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
  - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☒ has been transmitted by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)).
  - a. ☐ are transmitted herewith (only if not required by the International Bureau).
  - b. ☐ have been transmitted by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☐ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

**Items 11 to 16 below concern document(s) or information included:**

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ As assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
14. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
15. ☐ A substitute specification.
16. ☒ A change of power of attorney and/or address letter.
17. ☒ Other items or information:  
Application Data Sheet



23338

PATENT TRADEMARK OFFICE

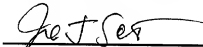
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17. <input checked="" type="checkbox"/> The following fees are submitted:				<b>CALCULATIONS</b> PTO USE ONLY	
<b>BASIC NATIONAL FEE (37 CFR 1.492 (a)(1)-(5):</b>					
Neither international preliminary examination fee (37 CFR 1.482)					
Nor international search fee (37 CFR 1.445(a)(2) paid to USPTO					
And International Search Report not prepared by EPO or JPO..... \$1,000.00					
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by EPO or JPO.....\$860.00					
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International search fee (37 CFR 1.445(a)(2)) paid to USPTO..... \$710.00					
International preliminary examination fee paid to USPTO (37 CFR 1.482) But all claims did not satisfy provisions of PCT Article 33(1)-(4).....\$690.00					
International preliminary examination fee paid to USPTO (37 CFR 1.482) And all claims satisfied provisions of PCT Article 33(1)-(4)..... \$100.00					
<b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b>				\$860.00	
Surcharge of \$130.00 for furnishing oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e).				\$	
<b>CLAIMS</b>	<b>NUMBER FILED</b>	<b>NUMBER EXTRA</b>	<b>RATE</b>		
Total Claims	6 -20=		X \$18.00	\$	
Independent Claims	1 -3=		X \$80.00	\$	
<b>MULTIPLE DEPENDENT CLAIM(S) (if applicable)</b>				\$	
<b>TOTAL OF ABOVE CALCULATIONS =</b>				\$860.00	
Reduction of 1/2 for filing by small entity, if applicable. A Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28).				\$	
<b>SUBTOTAL =</b>				\$860.00	
Processing fee of \$130.00 for furnishing English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f).				\$	
<b>TOTAL NATIONAL FEE =</b>				\$860.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31).				\$	
<b>TOTAL FEES ENCLOSED =</b>				\$860.00	
				Amount to be refunded:	\$
				charged:	\$

- a. ☐ A check in the amount of \$ to cover the above fees is enclosed.
- b. ☐ Please charge my Deposit Account No. 04-0753 in the amount of \$ to cover the above fees. A duplicate copy of this sheet is enclosed.
- c. ☐ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 04-0753. A duplicate copy of this sheet is enclosed.
- d. ☒ A payment of \$ 860.00 is made by credit card. A Credit Card Payment Form (PTO-2038) is attached hereto. The Commissioner is hereby authorized to charge payment of any additional filing fees required under 37 CFR 1.16 or any patent application processing fees under 37 CFR 1.17, or credit any over payment to the credit card account shown on the attached Credit Card Payment Form. Refund of all amounts overpaid, including those of twenty-five dollars or less, is specifically requested. Any fees not accepted by the credit card shown on Form PTO-2038 may be charged to Deposit Account No. 04-0753.

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

BENOIT CRISTOL

PCT

Serial No.: none assigned  
(PCT/FR99/01975)

Filed: Concurrently Herewith

For: PROCESS FOR CONTROLLING THE PARTICLE SIZE  
IN A BAYER CIRCUIT PRECIPITATION SYSTEM,  
INCLUDING AN AGGLOMERATION PHASE

PRELIMINARY AMENDMENT AND INFORMATION DISCLOSURE STATEMENT

Honorable Commissioner of Patents and Trademarks  
Washington, D.C. 20231

Sir:

Before calculation of the filing fee, please amend  
the above-identified application as follows:

IN THE CLAIMS:

Page 20, above line 1, change "CLAIMS" to --WHAT IS  
CLAIMED IS:--

Claim 4, line 2, change "one of claims 1 to 3" to  
--claim 1--.

Claim 5, line 2, change "any one of claims 1 to 4"

to --claim 1--.

Claim 6, line 2, change "one of claims 1 to 5" to  
--claim 1--

REMARKS

The claims have been amended to delete multiple dependencies.

Attached hereto is the Search Report of the corresponding PCT application, together with copies of the references cited therein, which are listed on the attached Form PTO-1449.

Respectfully submitted,



Ira J. Schultz  
Registration No. 28666

PROCESS FOR CONTROLLING THE PARTICLE SIZE IN A BAYER  
CIRCUIT PRECIPITATION SYSTEM, INCLUDING AN  
AGGLOMERATION PHASE

Technical domain

The invention relates to the precipitation of alumina trihydrate according to the Bayer process, carried out in an American type precipitation system including a preliminary agglomeration phase.

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State of prior art

The Bayer process can produce alumina from bauxite ore, particularly alumina designed to be transformed into aluminum by igneous electrolysis. According to this process, the bauxite ore is treated when hot by means of an aqueous sodium hydroxide solution with an appropriate concentration in order to obtain a suspension containing pregnant sodium aluminate liquor and insoluble residues. After separation of these residues, the pregnant sodium aluminate liquor, also called Bayer liquor, is decomposed by seeding with recycled aluminum trihydroxide particles until aluminum trihydroxide grains (also called alumina trihydrate or hydrargillite) are obtained, and which are themselves then calcinated to obtain an alumina with particular particle size distribution and physicochemical properties. The sodium aluminate liquor depleted in alumina (spent liquor) is then recycled to digest the ore, possibly after being concentrated, by the evaporation and addition of sodium hydroxide or caustic soda.

The productivity of the liquor during its crystallisation is defined by the quantity of alumina restored in the form of alumina trihydrate by crystallisation of the pregnant liquor, related to a given volume of pregnant liquor. The productivity expressed in kilograms of alumina per cubic meter of liquor ( $\text{kg of Al}_2\text{O}_3/\text{m}^3$ ), is related to the caustic concentration in the pregnant liquor. In general, this concentration in American type Bayer processes is close to 100-130 g of  $\text{Na}_2\text{O}$ / liter, which is lower than in European type Bayer processes, and this explains why a productivity in the crystallisation of the pregnant liquor is considered to be good when it exceeds 70  $\text{kg of Al}_2\text{O}_3/\text{m}^3$  for an American type Bayer process, or when it exceeds 80  $\text{kg of Al}_2\text{O}_3/\text{m}^3$  for a European type Bayer process.

The difference between European type and American type Bayer processes is in the solid content of the slurry during precipitation. The slurry is the result of introducing a recycled alumina trihydrate seed into the aluminate liquor, and part of the alumina in solution changing to the solid phase. We will define the solid content in the slurry as the weight of solid particles present in the slurry per unit volume of pregnant aluminate liquor entering into the precipitation workshop (and not per unit volume of the suspension).

Alumina to be transformed into aluminum by igneous electrolysis must have a number of properties, including:

- good flowability so that electrolysis tanks can be continuously supplied with controlled quantities of alumina,

- a high dissolution rate,
- a low tendency to dusting.

These properties are closely related to the morphology and the particle size distribution of alumina grains, themselves closely related to the morphology and particle size distribution of hydrargillite particles formed during the precipitation. It is particularly important to limit the proportion of very fine particles that can be classified in two main categories: fines (for which the average diameter is between 10 and 50  $\mu\text{m}$ ) and ultrafines, for which the average diameter is less than 10  $\mu\text{m}$ . Since a good correlation is observed between the particle size distribution of alumina and the particle size distribution of the production hydrate from which it is derived, an attempt is made to control the particle size of the rotating hydrate used in the precipitation series, and particularly in the crystal growth phase. Thus for example, in order to obtain a good quality "metallurgical" alumina, an attempt is made to obtain a rotating hydrate for which the amount passing a 45  $\mu\text{m}$  sieve is less than 10%, in other words a suspension containing less than 10% of particles with a diameter of less than 45  $\mu\text{m}$ . In the rest of this discussion, we will denote the quantity passing X micrometers as "%<X".

Concerning the American type Bayer process, the precipitation comprises a preliminary agglomeration phase characterized by a particularly low solid content. In patent US 4 234 559, the precipitation circuit comprises firstly a series of agglomeration tanks and then a series of feed tanks and finally three classification tanks (primary, secondary, tertiary).

While the hydrate produced is derived from the underflow from the primary classification tank, the fines seed originating from the underflow from the tertiary classification tank is inserted in controlled quantities into the series of agglomeration tanks and the larger seed originating from the underflow from the secondary classification tank is added into the series of feed tanks. Since the fines are destroyed during the agglomeration phase, the problem of controlling the particle size of the hydrate produced does not arise.

But attempts are always being made to increase the productivity of the American type Bayer process, which is lower than the productivity of European type Bayer cycles. In US 5 158 577 and EP 0 515 407, only part of the pregnant liquor is added into the agglomeration system, and the rest is added directly to the crystal growth. This can give different solid contents in these two parts of the crystallization, a low content of dry material in agglomeration tanks which is essential if it is required that agglomeration should take place under good conditions, and a high solid content in feed tanks which can increase productivity.

But an instability in the precipitation is observed if the content of dry material is increased at the feed and if the number of agglomeration tanks is limited, with a serious risk of the sudden appearance of large quantities of fines in the rotating hydrate (particle size crisis). This type of crisis should be avoided, since if it is not corrected the particle size quality of the produced hydrate is strongly deteriorated.

The particle size instability is due to a reduction in the ratio between the sum of the production and the fine seed sent to agglomeration, and the rotating



hydrate. A reduction in this ratio makes it impossible to implement effective corrective actions when a drift of the amount passing 45  $\mu\text{m}$  ( $\% < 45$ ) is observed on the rotating hydrate.

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#### Statement of the problem

Therefore, the applicant attempted to define a process that could increase the productivity of the American type Bayer process by preventing unacceptable particle size fluctuations, particularly the sudden appearance of large quantities of fines and ultrafines in the rotating hydrate.

#### Purpose of the invention

The process developed by the applicant is a process for controlling the precipitation of an American type Bayer circuit including a preliminary agglomeration phase, a crystal growth phase and a classification phase, in which the particle size quality of the hydrate produced is monitored by measuring the amount of rotating hydrate passing X2  $\mu\text{m}$  in feed tanks, characterized in that it comprises:

- a) a preparation step carried out once and for all, intended firstly to setup a relation R in intensity and in time between rotating hydrate material passing X1  $\mu\text{m}$  and material passing X2  $\mu\text{m}$ , where X1 is less than X2, and secondly to define trigger thresholds on the value of material passing X1  $\mu\text{m}$ , starting from the maximum authorized variation interval on values passing X2  $\mu\text{m}$ ;

- b) control of the process itself, carried out during the installation operating period which, apart from the daily measurement of material passing  $X2 \mu\text{m}$  and a regular update of the correlation between the said material passing  $X2 \mu\text{m}$  and the particle size of the hydrate produced, a daily measurement of the rotating hydrate passing  $X1 \mu\text{m}$  and a regular update of the relation R between the said material passing  $X1 \mu\text{m}$  and the said material passing  $X2 \mu\text{m}$ , and triggering of corrective action in the slurry at the beginning of the precipitation when the measured value of material passing  $X1 \mu\text{m}$  reaches one of the regularly updated trigger thresholds determined in the previous step.

This corrective action in the slurry at the beginning of precipitation may be a modification to the temperature of the aluminate liquor added into the agglomeration tank, the addition of additives at the beginning of the crystal growth system such as "Crystal Growth Modifiers" described in US 4 737 352, recycling a part of the end of crystallisation slurry, or preferably, modification of the solid content in the slurry in the first agglomeration tank.

- The solid content in the slurry in the agglomeration phase may be modified simply by adding more or less pregnant aluminate liquor in the first agglomeration tank, the remaining aliquot being directed to the feed tank. If the quantity of material passing  $X1 \mu\text{m}$  is too great, there are too many fines; the amount of pregnant aluminate liquor fed into the agglomeration tank must be increased. If the amount passing  $X1 \mu\text{m}$  is too low, there is a risk that

productivity will drop; the pregnant aluminate liquor feed from the agglomeration tank needs to be reduced.

The preliminary step, carried out once only, is intended to determine the relation  $R$  and trigger thresholds for the values of  $X1 \mu\text{m}$  that will be used at the beginning of application of the process control. This preliminary step comprises the following steps:

- 10 a1) Daily measurement of material passing  $X1 \mu\text{m}$  in the slurry at a particular point in the precipitation system, which is used to produce a first particle size time diagram represented by a curve  $Y = \%<X1(t)$ .
- 15 a2) Daily measurement of material passing  $X2 \mu\text{m}$  in the slurry at a particular point in the precipitation system, which is used to produce a second particle size time diagram represented by a curve  $Y = \%<X2(t)$  and in which  $X2$ , greater than  $X1$ , is a value already known for its good correlation with the particle size of the hydrate produced. For example, it may be material passing  $45 \mu\text{m}$  measured in slurry at pump-off.
- 20 a3) Creation of an empirical relation between the two particle size time diagrams, the purpose of which is to characterize the relation between the variation of the population of material passing  $X1 \mu\text{m}$  and the variation of the population of material passing  $X2 \mu\text{m}$ , in intensity and in time. This relation  $R$  may be written in the form:
- 25
- 30

$$R(\%<X2(t), \%<X1(t-\tau)) = 0$$

where  $t$  is the date on which material passing  $X2 \mu\text{m}$  is measured and  $\tau$  is a characteristic

time interval estimated by observing the occurrence of the same accidental phenomenon on each curve (the same type of extreme, an inflection, etc.).

- 5 a4) Definition of the maximum threshold and the minimum threshold of material passing  $X_1 \mu\text{m}$ , obtained from the relation R previously established and a maximum interval of the authorized variation of values of material  
10 passing  $X_2 \mu\text{m}$ .

The purpose of the relation R is to predict the variation in the particle size, in other words to anticipate crises, by observing the variation in the population of the finest hydrate particles (with a size  
15 of less than  $X_1 \mu\text{m}$ ). The applicant observed that material passing  $X_1 \mu\text{m}$  anticipates a change in the material passing a higher  $X_2$  value; an accident on the time diagram  $\%<X_1(t)$  is amplified and shifted in time on the  $\%<X_2(t)$  time diagram. The time shift " $\tau$ " is  
20 higher as the difference between  $X_1$  and  $X_2$  increases. In practice, a value  $X_2$  greater than  $40 \mu\text{m}$  (normally  $45 \mu\text{m}$ ) will be chosen, and the value of  $X_1$  will be taken to be less than or equal to  $20 \mu\text{m}$ .

The measurement point is preferably at pump-off,  
25 but it may take place earlier, provided that it remains within the crystal growth series. Measurement points for material passing  $X_1$  and for material passing  $X_2$  may be different. However, they must remain the same throughout the process control and must be as far as  
30 possible from the points at which disturbing additions are made irregularly in the slurry.

Measurements referred to as being "daily" are regular measurements and although they are not

necessarily daily, they are sufficient frequent to give useable time diagrams.

Concerning the required variation interval of material passing  $X_2 \mu\text{m}$ , a maximum is defined above which it is known that the particle size of the hydrate produced is no longer satisfactory (too many fines) and a minimum is defined below which it is known that economic operating conditions become bad.

The trigger thresholds are thus determined from the time diagram for the material passing  $X_1 \mu\text{m}$ , taking account not only of the maximum authorized variation interval on the values of material passing  $X_2 \mu\text{m}$ , but also the uncertainty of the measurement of material passing  $X_1 \mu\text{m}$  and the stability of the efficiency of the hydrate classification system.

This preparatory step, at the end of which the time diagrams, the relation R and the trigger threshold become operational, is of the order of three months. But this step can be accelerated either by deliberately triggering an excess creation of fines, or by analyzing previous particle size results if they contain the required information about material passing  $X_1$  and  $X_2 \mu\text{m}$ .

Since precipitation is a complex phenomenon that depends on a large number of parameters (particularly the composition of the treated bauxite that may change in time), instead of using a relation R established once and for all, it is better to use a relation that is regularly updated. Similarly, the correlation between the value of material passing  $X_2 \mu\text{m}$  and the particle size of the hydrate produced must be updated regularly. This regular updating may be applied at a

less frequent rate than daily measurements (for example monthly).

The actual process control comprises the following phases:

- 5        b1) A daily measurement of the material passing  
 $X1 \mu\text{m}$  in the slurry at a particular point in  
 the precipitation system, in order to complete  
 the first particle size time diagram  
 represented by the curve  $Y = \%X1(t)$ .
- 10       b2) A daily measurement of the material passing  
 $X2 \mu\text{m}$  in the slurry at a particular point in  
 the precipitation system, in order to complete  
 the first particle size time diagram  
 represented by the curve  $Y = \%X2(t)$ .
- 15       b3) Regular updating (for example monthly) of the  
 empirical relation  $R$  between the two particle  
 size time diagrams and the definition of  
 trigger thresholds of material passing  $X1 \mu\text{m}$ ,  
 or updating after an important modification in  
 a process parameter.
- 20       b4) Triggering of a corrective action in the slurry  
 at the beginning of the precipitation when the  
 measured value of material passing  $X1 \mu\text{m}$   
 reaches one of the thresholds defined in b3).
- 25       This corrective action is preferably a modification  
 to the solid content in the slurry in the first  
 agglomeration tank. If it is a maximum threshold,  
 there are too many fines, the content of dry material  
 is reduced by introducing a stronger aliquot of the
- 30       pregnant aluminate liquor into the first agglomeration  
 tank. If it is a minimum threshold, the feed of  
 pregnant aluminate liquor into the agglomeration tank

is reduced and the crystal growth into feed tanks is increased.

- As for the preparatory step, empirical relations are determined starting from daily particle size measurements made on the rotating hydrate. As for the preparatory step, the daily rate is not necessarily daily, but is sufficiently frequent to be able to produce useable time diagrams. Trigger thresholds on the "X1  $\mu\text{m}$  passing material" curve are also deduced from the particle size time diagram; they take account of the uncertainty of the measurement of material passing X1, the maximum authorized variation interval on values of material passing X2  $\mu\text{m}$ , and the stability of the efficiency of the hydrate classification system.
- For relatively low solid content ( $\approx 350$  g of aluminate/ liter), it is sufficient to measure amount passing X1 = 20  $\mu\text{m}$ . This may be done using instruments that diffract laser beams and make mass determinations (Malvern Mastersizer, Cilas, etc.).
- But in order to increase productivity, much higher solid content must be achieved, comparable to those achieved in European type processes, in other words greater than 700 g of aluminate/ liter. However, as the solid content in feed tanks increases, the need for a low value of X1 also increases. When the solid content is high, a longer time is necessary to correct particle size disturbances; therefore, it is necessary to have a greater time shift  $\tau$ ; and this time shift is proportional to the difference between X1 and X2.
- In this case, it is preferable to measure the amount passing 10  $\mu\text{m}$ , or even a lower value (down to 1.5  $\mu\text{m}$ ). When X1 becomes this low, it becomes necessary to use a celloscopic measurement (COULTER or

ELZONE) that determines counts rather than mass. These measurements are more difficult, but the economic consequences are negligible.

Productivity can also be increased by increasing  
5 the caustic content of the aluminate liquor. The applicant has observed that this method of controlling the particle size can be applied very well on industrial installations with concentrations reaching 160 g of  $\text{Na}_2\text{O}$ / liter.

10 Concerning the corrective action, it is also recommended that an empirical relation should be set up in advance to quantify the effects of the said action, for example during the preparatory phase. Thus, in order to quantify the effects of the modification to  
15 the solid content in the first agglomeration tank, the first step will be to establish the actual relation between the said solid content and the proportion of destroyed fines.

20 Embodiments of the invention - Examples

The embodiment of the invention will be better understood from the following description of a number of examples.

25 Figures 1 and 2 show the part of the Bayer circuit corresponding to the precipitation phase, the precipitation being of the American type with a preliminary agglomeration step.

Figure 1 illustrates the first example typical of  
30 prior art.

Figure 2 illustrates subsequent examples describing two embodiments of this invention.



These embodiments are described in examples 2 and 3. To simplify the presentation, we have illustrated the classification phase with all conventional classification devices in example 1. In fact, and particularly with the increase in the solid content, an expert in the subject could select additional or replacement systems such as cyclones or filters.

#### Example 1 (Prior art)

10

The pregnant aluminate liquor 1 enters the precipitation circuit at a temperature of about 75°C, enters the first agglomeration tank A. It has a caustic concentration of 130 g of Na2O/ liter.

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The tertiary seed 9, composed essentially of fine and ultrafine particles, is added into the first agglomeration tank A and is mixed with pregnant liquor. The resulting slurry 2 passes through a sequence of agglomeration tanks such that practically all fines and ultrafines have disappeared after an average residence time of about 5 hours. During the agglomeration phase, the solid content in the slurry has increased from 20 g of aluminate / liter to about 30 g of aluminate / liter.

20

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At the exit from the agglomeration series, the slurry 3 is added into the first feed tank N with the secondary seed 8, and the addition increases the solid content by 80 g of aluminate / liter. The new slurry 4 passes through a sequence of feed tanks with an average residence time of about 20 hours, and cooling by about 5°C. At the exit from the feed, the slurry 5, called the "pump-off", contains about 175 g of dry aluminate material / liter. By measuring the amount of this

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slurry 5 passing 45  $\mu\text{m}$  at the exit from the crystal growth **M1**, the particle size quality of the production hydrate can be estimated so that an optimum variation interval can be defined for this material passing  
 5 45  $\mu\text{m}$ . Under the conditions in the example, approximately 10% of material passing 45  $\mu\text{m}$  will give a good quality "metallurgical" alumina.

The slurry 5 at the exit from the crystal growth is then added into the first classifier tank **PT**. The  
 10 overflow 100 from the first classifier **PT** produces production hydrate and the overflow 6 that has a solid content of 100 g of aluminate / liter, is added into a second classifier tank **ST**. The underflow from the second classifier tank **ST** acts as a secondary seed 8  
 15 that is reinjected at the beginning of the crystal growth **N** and the overflow 7, which then has a solid content of only 20 g/ liter, is sent to a third classifier tank **TT**. The overflow 10 from the third classifier tank is the spent aluminum liquor that is  
 20 reinjected as the bauxite green liquor at the beginning of the Bayer cycle, after concentration by evaporation and the addition of soda. The overflow 9 from the third classifier tank is added back as the tertiary seed into the first agglomeration tank **A** after  
 25 filtration and washing.

**Example 2:** Precipitation according to the invention with an average solid content in the rotating hydrate

30 The pregnant aluminate liquor 1 that enters the precipitation circuit at a temperature of about 75°C, is separated into two aliquots, the first aliquot 1a

representing about one third of the total liquor being added into the first agglomeration tank **A**, and the second aliquot **1n** being added into the first feed tank **N**. The caustic concentration in the pregnant aluminate liquor **1** is 130 g of Na<sub>2</sub>O/ liter.

The tertiary seed **9a**, composed essentially of fine and ultrafine particles, is added into a first agglomeration tank **A** after filtration and partial or complete washing, and is mixed with the pregnant liquor. The resulting suspension **2** follows a sequence of agglomerating tanks such that a predefined proportion of fines and ultrafines has disappeared after an average residence time of 5 hours. During the agglomeration phase, the quantity of hydrate is increased by 10 g of aluminate / liter entering the workshop (1a and 1n).

The slurry **3** at the exit from the agglomeration series is added into the first feed tank **N** with the second aliquot **1n** of pregnant aluminate liquor, the rest of the washed or unwashed seed **9n**, and the secondary seed **8**, the addition of which increases the solid content by 220 g of aluminate / liter. The new slurry **4** follows a series of feed tanks with an average residence time of 20 hours and cooling of 5°C. At the exit from the crystal growth, the slurry **5** contains about 350 g of dry aluminate material / liter.

The slurry **5** at the exit from the crystal growth is added into a first classifier tank **PT**. The underflow **100** from the first classifier **PT** supplies the production hydrate, and the overflow **6** which has a solid content of 250 g of aluminate / liter is added into a second classifier tank **ST**. The underflow from

the second classifier tank **ST** acts as a secondary seed **8** that is reinjected at the beginning of the crystal growth **N** and the overflow **7**, which then only has a solid content of 30 g/ liter, is sent to the third classifier tank **TT**.

A measurement **M1** of the material passing 45  $\mu\text{m}$ , and a measurement **M2** of the material passing 20  $\mu\text{m}$ , are made in the slurry every day at pump-off, using a laser diffraction apparatus.

Observations of the particle size quality of the hydrate produced can be used to define the authorized variation interval on values of material passing 45  $\mu\text{m}$ , and the target set value **C** on the material passing 20  $\mu\text{m}$  is defined, by means of the empirical relation defined during the preparation phase and continually updated afterwards.

Depending on the difference between **M2** and the value of the set value **C** defined in advance to guarantee the particle size quality of the product and the differences **M2 - C** obtained in the previous days, the quantity of fines to be agglomerated in addition to or less than the previous day, are determined.

During the preparation phase, the relation between the solid content in the agglomeration phase and the proportion of destroyed fines was determined. This relation is used to fix the aliquot **1a** used in the agglomeration. This aliquot **1a** fixes the solid content in the agglomeration tanks and therefore the change required for destruction of the fines.

Furthermore, starting from measurements **M1** and **M2** made on previous days, the relation between the set value **C** and the required level **M1** for the material passing 45  $\mu\text{m}$ , and secondly the relation between the

solid content and fines destroyed in the agglomeration, are adjusted.

**Example 3: Precipitation according to the invention**  
**5 with a high solid content in the rotating hydrate**

The pregnant aluminate liquor **1** arriving in the precipitation circuit at a temperature of about 75°C, is separated in two aliquots, the first **1a** representing  
 10 about half of the total liquor being added into the first agglomeration tank **A**, the second **1n** being added into the first feed tank **N**. The caustic concentration in the pregnant liquor **1a** is 130 g of Na<sub>2</sub>O/ liter.

The filtered tertiary seed **9**, composed essentially  
 15 of fine and ultrafine particles, is added into the agglomeration step **A** and mixed with the fraction **1a** of pregnant liquor. The resulting slurry **2** stays in the agglomeration phase for about 5 hours. The proportion between the quantity of tertiary seed and the flow of  
 20 liquor **1a** is adjusted such that a predefined proportion of fines and ultrafines disappears. The quantity of hydrate during the agglomeration phase is increased by 15 g of aluminate entering in the workshop / liter (**1a** and **An**).

25 At the exit from the agglomeration series, the slurry **3** is added into the first feed tank **N** and with the second aliquot **1n** of pregnant aluminate liquor and the secondary seed **8**, the addition of which increases the solid content by 840 g of aluminate / liter. The  
 30 new slurry **4** passes through a series of feed tanks with an average residence time of 18 hours and cooling by 10°C. At the exit from the crystal growth, the slurry

5 contains about 1000 g of solid aluminate content/ liter.

At least one measurement **M1** of the material passing 45  $\mu\text{m}$  is made every day at this location using a laser diffraction apparatus, and a measurement **M2** of the material passing 1.5  $\mu\text{m}$  is made with an ELZONE counter.

The slurry **5** at the exit from the crystal growth is added into a first classifier tank **PT**. The underflow **100** from the first classifier **PT** supplies the production hydrate and the overflow **6**, that has a solid aluminate content of 870 g/ liter, is added into a second classifier tank **ST**. The underflow from the second classifier tank **ST** is used as a secondary seed **8** that is reinjected at the beginning of the crystal growth **N** after filtration and the overflow **7**, that has a solid content that no longer exceeds 30 g/ liter, is sent to the third classifier tank **TT**.

The process for monitoring and controlling the particle size of the rotating hydrate in the slurry at pump-off is identical to that described in example 2.

Advantages of the process according to the invention

- Particle size fluctuations related to the increase in the inertia of the system originating from the increase in solid contents are avoided.
- The productivity of liquors can thus be increased:
  - by controlling crystallization at high solid contents without endangering the quality of the alumina produced,
  - by adjusting the pump-off particle size to the maximum level compatible with quality requirements and the production classification system.

CLAIMS

1. Process for controlling the precipitation of a BAYER circuit including a preliminary agglomeration phase (A), a crystal growth phase (N) and a classification phase (PT; ST; TT), in which the particle size quality of the hydrate produced is monitored by making a measurement (M2) of the amount of rotating hydrate in the feed tanks, characterized in that it comprises:
- a) a preparation step carried out once and for all, intended firstly to setup a relation R in intensity and in time between the rotating hydrate material passing  $X1\text{ }\mu\text{m}$  and material passing  $X2\text{ }\mu\text{m}$ , where  $X1$  is less than  $X2$ , and secondly to define trigger thresholds on the value of material passing  $X1\text{ }\mu\text{m}$ , starting from the maximum authorized variation interval on values passing  $X2\text{ }\mu\text{m}$ ;
  - b) control of the process itself, carried out during the installation operating period which, apart from the daily measurement (M2) of material passing  $X2\text{ }\mu\text{m}$  and a regular update of the correlation between the said material passing  $X2\text{ }\mu\text{m}$  and the particle size of the hydrate produced, a daily measurement (M1) of the rotating hydrate passing  $X1\text{ }\mu\text{m}$  and a regular update of the relation R between the said material passing  $X1\text{ }\mu\text{m}$  and the said material passing  $X2\text{ }\mu\text{m}$ , and triggering of corrective action on the slurry at the beginning of the precipitation when the measured value of material passing  $X1\text{ }\mu\text{m}$  reaches one of the



regularly updated trigger thresholds determined in the previous step.

2. Process for controlling the precipitation of a BAYER circuit according to claim 1, characterized in that the said corrective action includes modification of the solid content in the slurry at the beginning of the precipitation.

3. Process for controlling the precipitation of the BAYER circuit according to claim 2, in which the modification in the solid content in the slurry at the beginning of the precipitation is achieved by modifying the proportions of aliquots (1a) and (1b) of the pregnant aluminate liquor (1) feeding the first agglomeration tank (A) and the first feed tank (N), respectively.

4. Process for controlling the precipitation of a BAYER circuit according to one of claims 1 to 3, characterized in that  $X_2$  is greater than  $40\text{ }\mu\text{m}$  and  $X_1$  is less than  $20\text{ }\mu\text{m}$ .

5. Process for controlling the precipitation of a BAYER circuit according to any one of claims 1 to 4, characterized in that the measurements of material passing  $X_1\text{ }\mu\text{m}$  and  $X_2\text{ }\mu\text{m}$  are made on the slurry (5) at pump-off.

6. Process for controlling the precipitation of a BAYER circuit according to one of claims 1 to 5, characterized in that the concentration of pregnant aluminate liquor (1) is less than or equal to  $160\text{ g of Na}_2\text{O/ liter}$ .

PROCESS FOR CONTROLLING THE PARTICLE SIZE IN A BAYER  
CIRCUIT PRECIPITATION SYSTEM, INCLUDING AN  
AGGLOMERATION PHASE

ABSTRACT OF THE DISCLOSURE

Control over the precipitation of an American type BAYER circuit in which the particle size quality of the hydrate produced is monitored by making a measurement of the amount of rotating hydrate passing  $X2 \mu\text{m}$ ,

5 comprising:

a) a preparation step intended to setup a relation R between the material passing  $X1 \mu\text{m}$  and material passing  $X2 \mu\text{m}$ , where  $X1$  is less than  $X2$ , and then use R to deduce trigger thresholds on the amount of material passing  $X1 \mu\text{m}$ ;

10

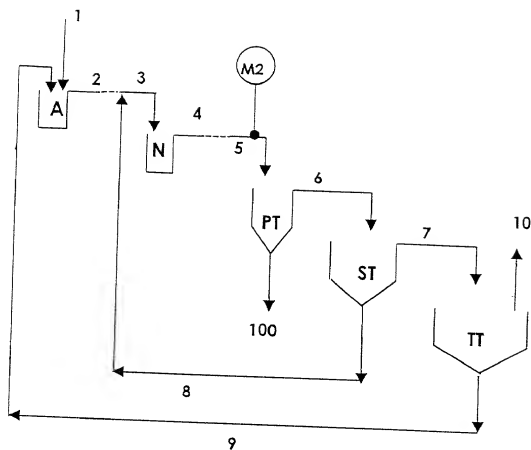
b) control of the process itself which, apart from the daily measurement of material passing  $X2 \mu\text{m}$ , includes a daily measurement of the rotating hydrate passing  $X1 \mu\text{m}$  and triggering of a corrective action in the slurry at the beginning of precipitation when the measured value of material passing  $X1 \mu\text{m}$  reaches one of the regularly updated trigger thresholds determined in the previous step.

15

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Figure 2.

- 1 / 2 -

Fig. 1

- 2 / 2 -

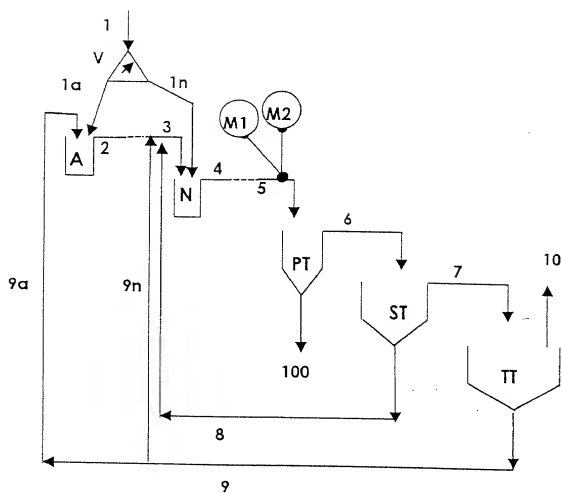


Fig. 2

# DECLARATION (37 CFR 1.63) FOR A UTILITY OR DESIGN APPLICATION USING AN APPLICATION DATA SHEET

As below named inventor(s), I/we declare that:

This application is directed to:

- ☐ The attached application; or  
☒ Application No. 09/744,237, filed on January 26, 2001,  
☒ as amended on January 26, 2001 (if applicable);

I/we believe that I/we am/are the original and first inventor(s) of the subject matter which is claimed and for which a patent is sought;

I/we have reviewed and understand the contents of the above-identified application, including the claims, as amended by any amendment specifically referred to above;

I/we acknowledge the duty to disclose to the United States Patent and Trademark Office all information known to me/us to be material to patentability as defined in 37 CFR 1.56, including material information which became available between the filing date of the prior application and the National or PCT International filing date of the continuation-in-part application, if applicable; and

All statements made herein of my/our own knowledge are true, all statements made herein on information and belief are believed to be true, and further, that these statements were made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001, and may jeopardize the validity of the application or any patent issuing thereon.

## FULL NAME OF INVENTOR(S)

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Signature: 

Date: 15/02/2001

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Date: \_\_\_\_\_

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Date: \_\_\_\_\_

Inventor four: \_\_\_\_\_

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Signature: \_\_\_\_\_

Date: \_\_\_\_\_

☐ Additional inventor(s) is/are being named on additional form(s) attached hereto.

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09/744237  
500 Rec'd PCT/PTO 2 6 JAN 2001

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## APPLICATION INFORMATION

Title Line One:: PROCESS FOR CONTROLLING THE PARTICLE SIZE  
Title Line Two:: E IN A BAYER CIRCUIT PRECIPITATION SYSTEM  
Title Line Three:: M, INCLUDING AN AGGLOMERATION PHASE  
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Docket Number:: 01006  
Secrecy Order in Parent Appl.?: No

## REPRESENTATIVE INFORMATION

Registration Number One:: 19920  
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Registration Number Four:: 37750

## CONTINUITY INFORMATION

This application is a:: 371 OF  
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Patent Number:: WO00/14015

## PRIOR FOREIGN APPLICATIONS

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Priority Claimed:: Yes

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